भारतीय मानक

IS 18465 : 2023

Indian Standard

पेट्रोकेमिकल के लिए फीडस्टॉक के रूप में उपयोग के लिए ईथेन — विशिष्टि

Ethane for Use as Feedstock for Petrochemical — Specification

ICS 75.080

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भारतीय मानक ब्यूरो
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November 2023 **Price Group 8**

Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee, PCD 03

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The purpose of this standard is to provide the information on the quality parameters for ethane used in petrochemical processing. Ethane is present as a constituent of natural gas and it is also produced in refineries through cracking processes. Ethane is mainly used to produce ethylene, which is then used by the petrochemical industry to produce a variety of intermediate products, most of which are converted into polymers like polyethylene, mono ethylene glycol (MEG), etc.

Ethane is highly flammable and safety issues are relevant. Ethane should be kept away from heat, hot surfaces, sparks, open flames and other ignition sources. It may also displace oxygen and cause suffocation.

Considerable assistance has been drawn from industrial specifications prevalent at this time. This standard is formulated based on the request received from Department of Chemicals and Petrochemicals (DCPC).

The composition of Committee responsible for the formulation of this standard is given in Annex E.

For the purpose of deciding whether a particular requirement of this standard is compiled with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this standard.

Indian Standard

ETHANE FOR USE AS FEEDSTOCK FOR PETROCHEMICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for ethane used in petrochemical industries.

2 REFERENCES

The standards given below contain provisions which through reference in this test constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouragedto investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water (*fourth revision*)
IS 1447 (Part 2) : 2013/ ISO 4257 : 2001	Methods of sampling of petroleum and its products: Part 2 Liquefied petroleum gases — Method of sampling (*second revision*)
IS 1448	Methods of test of petroleum and its products:
(Part 151) : 2004/ ISO 7941 : 1988	Commercial propane and butane — Analysis by gas chromatography
(Part 152) : 2004/ ISO 6521 : 1996	Liquefied petroleum gases — Corrosiveness to copper — Copper strip test

3 TERMINOLOGY

3.1 Ethane — A gaseous hydrocarbon having chemical formula C_2H_6.

4 REQUIREMENTS

4.1 Ethane shall be free from liquids.

4.2 The material shall also comply with the requirements given in Table 1, when tested in accordance with test methods specified in col (4) of the Table 1.

5 STORAGE AND SUPPLY OF ETHANE

Ethane shall be stored and supplied in accordance with the local regulations. Compliance to regulatory provisions by the statutory authorities in this respect shall be mandatory.

6 SAMPLING

The representative samples of ethane shall be drawn as prescribed under IS 1447 (Part 2).

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water shall be used in tests (*see* IS 1070).

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of the analysis.

To access Indian Standards click on the link below:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knowyourstandards/Indian_standards/isdetails/

Table 1 Requirements of Ethane for Petrochemical Industries

(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Parts of IS 1448/Annex
(1)	(2)	(3)	(4)
i)	Composition:		Part 151
	a) Methane, percent (*v/v*), *Max*	0.5	
	b) Ethane, percent (*v/v*), *Min*	95.0	
	c) Ethylene and other C_2 hydrocarbons, percent (*v/v*), *Max*	1.0	
	d) C_3 and other heavier hydrocarbons, percent (*v/v*), *Max*	3.5	
ii)	Copper strip corrosion (40 °C for 1 h)	Not worse than 1	Part 152
iii)	Total sulphur content, ppm, *Max*	35	Annex A
iv)	Dew point, °C, *Max*	- 79	Annex B
v)	Carbon dioxide, ppm, *Max*	100	Annex C
vi)	Hydrogen sulphide, ppm, *Max*	2	Annex D
vii)	Carbonyl sulphide + CS_2, ppm, *Max*	2.5	Annex D

ANNEX A

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF TOTAL VOLATILE SULPHUR CONTENT IN ETHANE USING UV FLUORESCENCE DETECTOR

A-1 GENERAL

This method is used for the determination of total volatile sulphur content in ethane, using a UV fluorescence detector. In this procedure a sulphur chemiluminescence detector (SCD) is considered. The concentration range of detection will depend on the detector used, however it is applicable in the range of 1 mg/kg to 100 mg/kg.

A-2 OUTLINE OF THE METHOD

A-2.1 The gaseous sample is injected through a heated sample valve connected to a heated expansion chamber. The gaseous sample enters into a high temperature, dual temperature zone combustion tube wherein sulphur components are combusted and vaporized. The released sulphur is oxidized to Sulphur dioxide (SO_2) in an oxygen-rich atmosphere.

$$R — S + O_2 \xrightarrow{1\,050\,°C} CO + SO_2 + H_2O$$

A-2.2 A stream of inert gas (argon or helium) carries the reaction products after the produced water vapour is removed into a reaction chamber. In this chamber, the SO_2 molecules are converted to excited SO_2^* through the absorption of energy of a UV lamp and emitting light (fluorescence) while it settles into a stable state.

$$SO_2 + hv \rightarrow SO^*$$
$$SO_2 \rightarrow SO^* + hv$$

A-2.3 The emitted light signal is measured by a photomultiplier tube. To calculate the area, the response signal is integrated. The linear regression function of the concentration of standard mixtures versus integrated area can be used to calculate the sulphur concentration of an unidentified product.

A-3 APPARATUS

A-3.1 Furnace

An electric furnace, capable to maintain a temperature (1 050 °C ± 25 °C) to pyrolyze the entire sample and oxidize sulphur to SO_2.

A-3.1.1 *Combustion Tube*

A quartz combustion tube constructed to allow the direct injection of the sample into the heated oxidation zone of the furnace with provision for side arms for oxygen and carrier gas entry. The oxidation section shall be large enough to ensure complete combustion of the sample.

A-3.1.2 *Flow Control System*

The apparatus shall have four flow controllers to maintain a constant supply of oxygen and carrier gas at the specified rates (*see* **A-3.1.6**).

A-3.1.3 *Drier Tube*

The apparatus shall have mechanism (such as membrane drying tube, or a permeation dryer) for the removal of water vapor formed during sample combustion.

A-3.1.4 *UV Fluorescence Detector*

A quantitative detector capable of measuring light emitted from the fluorescence of sulphur dioxide by UV light.

A-3.1.5 *Sample Inlet System*

The system provides a heated gas-sampling valve, or a LP gas-sampling valve, or both, with a heated expansion chamber, connected to the inlet of the oxidation area. The system is swept byan inert carrier gas and shall be capable of allowing the quantitative delivery of the material to be analyzed into the oxidation zone at a controlled and repeatable rate of approximately 30 ml/min.

A-3.1.6 *Operational Conditions*

Sl No. (1)	*Parameter* (2)	*Specification* (3)
i)	Furnace temperature	1050 °C
ii)	Combustion time	60 s
iii)	Air flow (first phase)	200 ml/min
iv)	O_2 main flow	100 ml/min
v)	O_2 flow (second phase)	100 ml/min
vi)	Integration time, *Max*	300 s
vii)	Start	100 ppb
viii)	Threshold	100 ppb

A-4 REAGENTS

A-4.1 Inert Gas — argon or helium, high purity chromatography grade, 99.998 percent purity, *Min*, and moisture 5 mg/kg, *Max*.

A-4.2 Oxygen — high purity chromatography grade, 99.75 percent purity, *Min*, and moisture 5 mg/kg, *Max*.

A-4.3 Calibration Standards — certified calibration standards from commercial sources or calibration gases prepared using certified permeation tube devices shall be used.

A-5 PREPARATION OF THE EQUIPMENT

A-5.1 The calibration range shall be selected based on the expected sulphur concentration of the sample under evaluation. The standards shall be made using a sulphur compound as active ingredient with suitable non interfering diluent.

A-5.2 The calibration should be carried out using the manufacturer's instructions to yield sulphur concentration. The curve shall be linear and should meet the regression criteria of the manufacturer.

A-6 PROCEDURE

A-6.1 Connect to the sample point the needle valve (if required to reduce pressure) and allow the gas to enter the combustion chamber.

A-6.2 Flush the sample loop with the sample. Ensure sufficient sample has entered the loop tomake the measurement representing the sample. Measure the response for the test sample.

A-6.3 Each result shall be the average of at least three measurements.

A-7 CALCULATION

When the sample is injected in the liquid phase, the density of the calibration mixture and the sample are required to calculate the results. When the sample is injected in the gas phase, the molecular weight of the calibration mixture and the samples are required to calculate the results.

To calculate the density of the liquid calibration mixture and/or the sample:

$$\text{Liquid volume percent}_x =$$

$$100 \times \frac{\text{Mass percent}_x \,/\, \text{Relativedensity}_x}{\sum_{N=1}^{\#\,\text{comp}} \text{Mass percent}_n / \text{ Relative density}_n}$$

where

Liquid volume percent = liquid volume percent of all determined components in the sample of which x is one;

Mass percent$_x$ = the mass percent of each determined component taken from GC analysis;

Relative density$_x$ = the value given for each determined component x as a liquid; and

$\sum_{N=1}^{\#\,comp} Mass\ percent_n / Relative\ density_n$ = the sum of the quotients mass percent divided by the relative density for all determined components.

A-8 REPORT

Report the concentration of total volatile sulphur content to the nearest 1 ppm.

ANNEX B

[Table 1, *Sl No.* (iv)]

DETERMINATION OF DEW POINT

B-1 GENERAL

This method covers the determination of dew point (°C) of ethane as feedstock for petrochemical industries. Dew point is the temperature to which gas must be cooled for water vapor in it to condense into dew or frost.

B-2 OUTLINE OF METHOD

The dew point meter is connected to the sample point and a constant flow of sample is established. When a steady reading is obtained, the dew point of sample is read directly from the meter.

NOTES

1 Inflammable gas should be discharged to open air safely.

2 Avoid water condensation being drawn into sampling system and sensor of the meter. Condensationon the sensor will invalidate the readings and will take long time to dry out sample system.

B-3 APPARATUS

B-3.1 Dew Point Meter — preferably automatic and intrinsically safe or suitable equipment containing a sensor of desirable range of - 100 °C to 0 °C.

B-3.2 Needle Valve — to control the pressure, if required

B-3.3 Teflon or Stainless Steel Pipe Connections

B-4 CALIBRATION

Equipment should be calibrated as per

manufacturer's instructions.

B-5 PROCEDURE

B-5.1 Connect to the sample point the needle valve (if required to reduce pressure), the dewpoint meter and then the flow meter with stainless steel/teflon piping.

B-5.2 Ensure the sensor head on the meter is in the 'down' or in bypass position.

B-5.3 Open the needle valve and the valve on the flow meter, if present.

B-5.4 Carefully open the valves on the sample point to establish a flow of gas through the needle valve, moisture meter and flow meter.

B-5.5 Adjust the flow of the gas (desirably slow flow, 1 litre/min to 2 litre/min, control with the help of needle valve).

B-5.6 Allow sample to purge for a few minutes.

B-5.7 After sufficient purging of the lines switch on the dew point meter and allow the sample gas to pass through test chamber or sensor.

NOTE — If a high reading is obtained when switching on the meter with the sensor head down, then this would indicate the internal desiccant is contaminated with water and should be changed.

B-5.8 Allow the sample gas to flow through until a steady reading is obtained on the meter. Record the reading as dew point.

B-5.9 Push the sensor head back down so the sample gas is no longer in contact with the sensor.

B-5.10 Close all valves and disconnect from the sample point.

NOTE — Refer instrument manual or OEM instructions for operation.

B-6 REPORT

Report the dew point of the sample as recorded (*see* **B-5.8**).

NOTE — Moisture content can also be read as concentration, directly from the meter in ppm (v) or by referring to the moisture equivalent value chart, if supplied with equipment.

ANNEX C

[Table 1, *Sl No.* (v)]

DETERMINATION OF CARBON DIOXIDE IN ETHANE USING LENGTH OF STAIN DETECTOR TUBES

C-1 OUTLINE OF THE METHOD

The stain detector tubes contain chemical reagents that are designed to detect the presence of specific gases and display the concentration of these gases or vapors in the sample. The length of the resultant colour band in the tube indicates an instantaneous quantitative concentration of the specific chemical in the sample. The length of stain produced is converted to parts per million (ppm) or percent carbon dioxide by comparison to a calibration scale supplied by the manufacturer for each box of detection tubes. The system is direct reading, easily portable and completely suited to making rapid spot checks for carbon dioxide under field conditions.

C-2 APPARATUS

C-2.1 Pump and Tube Apparatus

C-2.1.1 A specially designed pump and colorimetric indicator tube detector (length-of-stain), with a supply of detector tubes, that operates by using the pump to pull a known volume of airor gas through a detector tube is used.

C-2.1.2 Length of stain detector tube sealed glass tube with break off tips sized to fit the tube holder of the pump. The reagent layer inside the tube, typically a silica gel substance coated with active chemicals, must be specific for carbon dioxide and produce a distinct colour change when exposed to sample containing carbon dioxide. Any substances known to interfere must be listed in the instruction accompanying the tubes. A calibration scale should be marked directly on the tube, however other marking that provide for easy interpretation of carbon dioxide content from a separate calibration scale supplied with tubes shall be acceptable.

C-2.2 The calibration scale shall correlate carbon dioxide concentration to the length of the colour stain. Shelf life of the detector tubes must be a minimum of two years from the date of manufacture when stored according to manufacturer's recommendation.

C-3 PROCEDURE

C-3.1 Test shall be carried out in duplicate.

C-3.2 Select a sampling point that provides access to representative sample of the gas being tested (source valve to main line). The sample point should be on top of the pipeline and equipped with a stainless steel sample probe extending into the middle third of the pipeline. Open the source valve momentarily to clear the valve and connecting nipple of foreign materials. Install needle valve (or pressure regulator) at the source valve outlet. Connect sampling chamber using the shortest length of flexible tubing possible (*see* Fig. 1). Most flexible tubing material will be suitable for carbon dioxide sampling. However, if the sampler is also used for other constituents such as hydrogen sulphide then choose tubing materials carefully. Avoid using tubing that reacts with or absorbs hydrogen sulphide, such as copper or natural rubber. Use material such as TFE-fluorocarbon, vinyl, polyethylene or stainless steel.

C-3.3 Open source valve. Open needle valve enough to obtain positive flow of gas through chamber. Purge the container for at least for 3 min. Before each series of measurements, the pump used must be tested for leaks.

C-3.4 Insert an unopened tube snugly into the inlet of the pump. Align the plunger on the pumpshaft. Pull the plunger one full stroke and wait 2 min. Rotate the plunger away from the pumpshaft, and allow the plunger to be drawn back into the pump shaft. Keep your hand on the shaftto keep it from snapping back too suddenly. There are no leaks if the plunger returns to within 3 mm of its original position. A leaking pump used in field testing will bias sample results low.

C-3.5 Select the tube range that best encompasses carbon dioxide concentration. Reading accuracy is improved when the stain length extends into upper half of the calibration scale.

NOTE — Consult manufacturer's guidelines for using multiple strokes to achieve a lower range on a given tube.

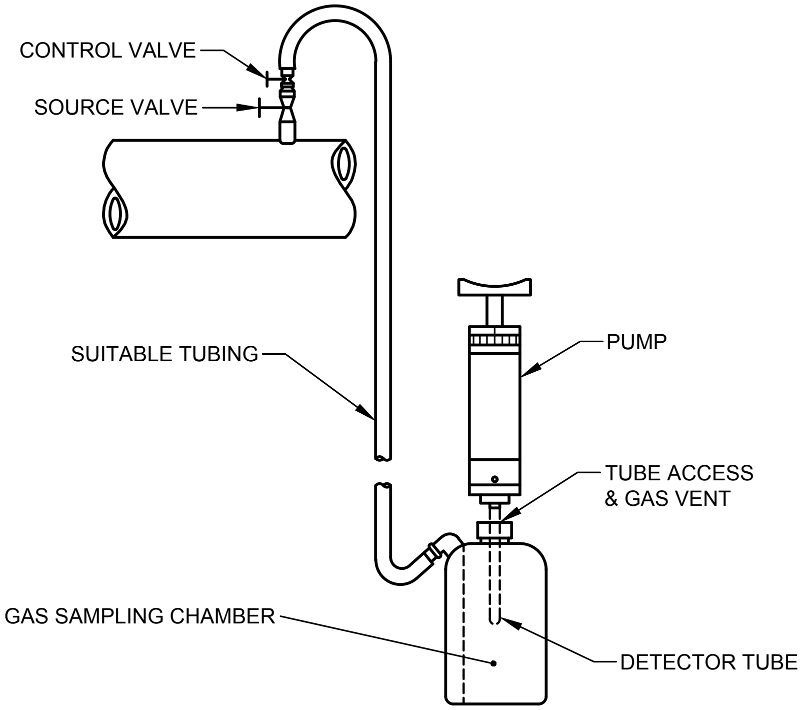

FIG. 1 PUMP AND TUBE APPARATUS

C-3.6 Break off the tube tips and insert the tube into the pump, observing the flow direction indication on the tube. Place the detector tube into the sampling chamber through the access hole. Such that the tube inlet is near the chamber centre.

C-3.7 Operate the pump to draw the measured sample volume through the detector tube. Observe tube instruction when applying multiple stroke. Ensure that a positive flow is maintained throughout the sample duration at the sampling chamber gas exit vent. Observe tube instruction for proper sampling time per pump stroke. The tube inlet must remain in position inside the sampling chamber until sample is completed. Many detector tube pumps have stroke finish indicator that eliminate the need to time the sample.

C-3.8 Remove the tube from the pump and immediately read carbon dioxide concentration from the tube’s calibration scale or from the charts provided in the box of tubes. Read the tube at the maximum point of the stain. If channeling has occurred (non-uniform length), read the maximum and minimum stain lengths and average the two. Consult tube instruction for any special information in the event of multicolour stains.

C-4 CALCULATION AND REPORT

C-4.1 If the number of strokes used differs from the number of strokes specified for the calibration scale correct the reading as follows:

$$ppm\ (corrected) = \frac{\text{ppm(reading)} \times \text{specified strokes}}{\text{actual strokes}}$$

C-4.2 Record the reading immediately along with the gas temperature and the barometric pressure. Observe any temperature corrections supplied in the tube instructions. Altitude corrections becomes significant at elevations above 2 000 feet. Correct for barometric pressure as follows:

$$ppm\ (corrected) = \frac{\text{ppm(reading)} \times \text{760 mm Hg}}{\text{baromatric pressure,in mm Hg}}$$

C-5 PRECISION

The accuracy of detector tube systems is generally considered to be ± 25 percent.

ANNEX D

[Table 1, *Sl No.* (vi) *and* (vii)]

DETERMINATION OF HYDROGEN SULFIDE (H_2S), CARBONYL SULFIDE (OCS) AND CARBON DISULFIDE (CS_2) IN ETHANE FEEDSTOCK USING GC AND SULPHUR SPECIFIC DETECTOR

D-1 GENERAL

This procedure prescribes the test method for the determination of trace levels of hydrogen sulfide (H_2S), carbonyl sulfide (OCS) and carbon disulfide (CS_2) in ethane, that is used as a feedstock, using a GC equipped with Sulphur specific detector. In this procedure a sulphur chemiluminescence detector (SCD) is considered.

The concentration range of detection will depend on the detector used, however it is applicable in the range of 0.1 mg/kg to 100 mg/kg for individual components.

D-2 OUTLINE OF THE METHOD

The sample is injected into a gas chromatographic system equipped with suitable separating column and the sulphur compounds are quantified by a sulphur specific detector capable of producing equimolar response to sulphur compounds.

D-3 APPARATUS

D-3.1 Gas Chromatograph

A gas chromatograph equipped with suitable sample inlet system, column oven with temperature programming, suitable sulphur specific detector and a data integrator.

D-3.1.1 *Sample Inlet System*

This system must be capable of variable temperature control and inject constant volume of sample using either a gas sampling valve (GSV) or liquid sampling valve (LSV). Additional facility of a split/splitless injection system would be required if a capillary column is used.

D-3.1.2 *Column*

Any column that can provide separation of the sulphur components of interest can be used. Capillary columns have better resolution. For the purpose of this procedure, a low sulphur, 60 m × 0.32 mm × optimized film thickness is considered. Any equivalent column can be used.

D-3.1.3 *Gas Control*

Must be capable of providing constant flow of carrier gas through column and detector. Pressure regulators, mass flow controllers are capable of providing such constant gas flows within ± 1 percent of the required flow rates.

D-3.1.4 *Detector*

Any sulphur selective detector can be used that can meet the requirements of linearity, minimum detectability and not quenching of other hydrocarbon components. For the purpose of this procedure, a sulphur chemiluminescence detector (SCD) is considered.

D-3.1.5 *Carrier Gas*

Helium or nitrogen of high purity can be used as a carrier gas. Filters and molecular sieve maybe used to remove oxygen, hydrocarbons and moisture.

D-3.1.6 *Detector Gases*

Hydrogen, nitrogen, air and oxygen may be required. These gases must be particularly free of any sulphur contamination.

D-3.1.7 *Calibration Standard*

Obtain a calibration mixture of carbonyl sulfide and carbon disulfide in ethane either in vapour phase or liquid phase as desired from a suitable producer who can supply the material with appropriate trace ability. Obtain the mixture in three different concentration levels that covers the desired level also.

D-4 PROCEDURE

D-4.1 As the modern gas chromatograph systems are equipped with software operating systems for methods preparation, parameters controls and data system follow the instructions as per the manufacturer’s recommendation.

D-4.2 Set the gas chromatograph conditions as mentioned in Table 2. These are typical values and the actual values may depend on the column and detector employed. Hence refer to the manufacturer's recommendations.

D-4.3 Calibration

Perform external calibration using the calibration mixtures. Run the standard and obtain at least three consecutive chromatograms with areas of components that are closely matching. Calculate the response factor for each sulphur component as below:

$$\text{Response factor} = \frac{\text{Concentration of component in standard}}{\text{Area of component}}$$

D-4.4 Analyse the sample the same way as the calibration mixture was run. Using the response factors calculate the concentration of the sulphur component. Identify the components by the retention times obtained which are specific to the test conditions adopted.

5 CALCULATION AND REPORT

D-5.1 The sulphur component and the matrix should be as per the sample stream's requirement. The same Matrix is preferred. Compare the area response of each sulphur compound of interest against external standard.

D-5.2 Calculate the concentration of each sulphur component using the below equation and report individual components to the nearest 0.1 ppm.

$$\text{Component concentration} = \text{Response factor by standard (RF)} \times \text{Area of component in sample}$$

Table 2 Typical Gas Chromatograph Conditions

(*Clause* D-4.2)

Sl No.	Parameter	Value
(1)	(2)	(3)
i)	Carrier gas	Helium
ii)	Inlet	Split/Splitless inlet in split
iii)	Inlet temperature	250 °C
iv)	Split ratio	2 : 1
v)	Septum purge flow	5 ml/min
vi)	Volume of sample loop (for gaseous sample)	500 µl
vii)	Column	Low sulphur, 60 m × 0.32 mm × optimized film
viii)	Column flow	2 ml/min
ix)	Column temperature program	40 °C for 1.5 min, 15 °C/min to 170 °C (hold 32 min)
x)	SCD conditions[1]	
a)	Top temperature	650 °C
b)	Bottom temperature	950 °C
c)	Base temperature	350 °C
d)	Hydrogen flow	Top: 130 ml/min Base: 12 ml/min
e)	Oxidant flow	100 ml/min
f)	Oxidant (O_3)	80 ml/min
g)	Base pneumatic control	25 kPa
h)	Cell vacuum	22 torr
j)	Furnace vacuum	220 torr

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[1] The actual operating conditions of the SCD depends on individual make. Refer and follow manufacturer's recommendations.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee, PCD 03

Organization	*Representative(s)*
Indian Oil Corporation (R and D Centre), Faridabad	Dr S. S. V. Ramakumar **(*Chairperson*)**
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Ashok Leyland Limited, Chennai	Shri Mahesh P. Shri Balakrishnan D. (*Alternate*)
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Petroleum Conservation Research Association, New Delhi	Shri Manoj Kumar Acharya Shri Manish Ranjan (*Alternate*)
Petroleum and Explosives Safety Organization, Nagpur	Shri S. K. Dixit Shri Ninad Gawade (*Alternate*)
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Indian Oil Corporation Limited - Refineries and Pipelines Division, New Delhi	SHRI S. BHAR SHRI CHANDAN PAL (*Alternate*)
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